THE NICKEL BULLETIN

A SUMMARY OF CURRENT INFORMATION ON NICKEL

NOVEMBER, 1952

Page

General

Nickel-Alloy Thermocouples: British Standard 256
Metallurgical Equilibrium Diagrams: Textbook 256

Nickel

Reduction of Nickel Oxide in a Fluidized Bed 256
Promotion of Nickel Catalysts 257
Nickel Kieselguhr 257
Recovery of Nickel from Silver-Refinery Waste Liquors 257
Determination of Small Amounts of Magnesium in Nickel 257
Chromatographic Determination of Nickel 257
Iodometric Titration of Nickel with Thiosulphate 257

Electrodeposition and Other Coating Methods

Properties of Electrodeposited Nickel: A.E.S. Report 258
Zinc in Nickel-Plating Solutions: Effects and Removal 258
Cast Nickel Anodes 259
'Udytal' Process of Direct Nickel Plating of Aluminium 259
Costing and Control of Materials in Nickel Plating 259
Silver Plating of Nickel-Iron Alloys 259
Adhesion Testing of Electrodeposited Metals: Modified 'Nodule' Method 260

Non-Ferrous Alloys

Induction-Furnace Melting of Nickel and Nickel Alloys 260
Low-Copper Nickel-Copper-Zinc Casting Alloy 261
High-Nickel Alloys used in Aircraft 262
Centrifugal Castings of Nickel-Brass and -Bronze: A.S.T.M. Specification 262
Hall Effect in Cobalt-Nickel Alloys 262
Elastic Properties of Nickel-containing Aluminium Alloys 262
Nickel-Rich Nickel-Titanium-Aluminium Alloys 262

Nickel-Iron Alloys

Influence of Temperature on Eddy-Current Loss in Thin Nickel-Iron Alloy Tape 263
Nickel-Iron-Molybdenum Ferromagnetic Alloy (Vibralloy) 263
Ultrathin Nickel-Iron Alloy Tape: Properties and Uses 263
Nickel-Alloy Magnetic Cores for Small Power Transformers 264
Nickel-Alloy Magnets in Switch Contacts 264
Magnetic Viscosity of Alnico under Variable Field Conditions 265
Magnetic Study of Nickel-Iron Alloy having Low Internal Stress 265

Page

Constructional Steels

Critical Temperatures for Plain and Alloy Steels: Data Sheet 265
Influence of Silicon on Tempering of Martensite in Nickel Steels 265
Influence of Various Forms of Heat-Treatment on Temper Brittleness 266
Effect of Hardness on Impact Energy in Temper-Brittle and Unembrittled Steel 267
Nickel-Chromium-Molybdenum Steel Mining Drill Rods: Fatigue Properties 267
Influence of Boron on Case-Hardenability of Nickel-Alloy Steels 267

Heat- and Corrosion-Resisting Alloys

High-Temperature Materials for Gas Turbines: Symposium 268
Creep-Rupture and Recrystallization Studies on Monel 268
Influence of Grain Size on High-Temperature Properties of Monel 268
High-Temperature Properties of Wrought Chromium-Nickel-Cobalt-Iron-base Alloys 269
Influence of Composition on Rupture Strength of Chromium-Cobalt-Nickel-Iron Alloys 269
Properties of Austenite and Sigma 270
Formation and Effect of Sigma Phase in Commercial Iron-Nickel-Chromium Alloys 270
Carburization of 18–8 Steels 271
Microconstituents in High-Temperature Alloys 271
Nimonic Alloys in Power-Station Gas Turbine 271
High-Temperature Properties of 18–8 Chromium-Nickel Steels 272
Resistance of Nickel-Chromium-Iron Alloy to Steam 272
Electron-Diffraction Investigation of Austenitic Nickel-Chromium Steel 272
Conservation of Nickel in Stainless Steels 272
Austenite-Martensite Transformation in Stainless Steels 273
Resistance of Metallic Materials to Attack by Hydrogen Peroxide 273
Corrosion-Resisting Nickel-Steel Tubing in Oil-Condensate Wells 273
Corrosion-Resisting Materials in Marine Engineering 273
Hastelloy 'D': Reference Data 274
Etching of Stainless Steel 274
Stainless-Steel Parts made by Powder Metallurgy 274
Testing of Corrosion-Resisting Materials 274

VOLUME 25 NUMBER 11

THE MOND NICKEL COMPANY LTD • SUNDERLAND HOUSE, CURZON STREET, LONDON.W.1
Telephone:- Grosvenor 4131

ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Nickel-Alloy Thermocouples: British Standard

BRIT. STANDARDS INSTN.: 'Reference Tables for Nickel-Aluminium v. Nickel-Chromium Thermocouples.' *B.S.* 1827: 1952.

This Standard, prepared under the aegis of the Instrument Industry Standards Committee, forms one of a series of reference tables for thermocouples. (Tables for platinum-rhodium v. platinum thermocouples are given in B.S. 1826: 1952. Tables for copper v. Constantan are in preparation.)

The electromotive-force/temperature tables for the nickel-alloy thermocouples given in this Standard are intended for conversion of thermocouple voltages into the equivalent measured temperatures, and also to define the relation between impressed e.m.f. and scale reading for pyrometers which indicate temperature directly. The tables are based on the International Temperature Scale, 1948, and on the Absolute Volt, which was adopted (in 1948) in place of the International Volt, as the standard unit.

The Standard specifies e.m.f.-temperature relations for the nickel-chromium v. nickel-aluminium thermocouples in four ways:—

Millivolts: °C. and °C.: millivolts.
Millivolts: °F. and °F.: millivolts.

The tables formulated by the National Bureau of Standards (as published in N.B.S. Circular 508, 1951) have been adopted, since no other tables are in common use in the United Kingdom.

Metallurgical Equilibrium Diagrams: Textbook

W. HUME-ROTHERY, J. W. CHRISTIAN and W. B. PEARSON: 'Metallurgical Equilibrium Diagrams.' Published by Inst. Physics, in *Physics in Industry* series, 1952; 311 pp. Price 50/-.

In spite of the interest which has been shown in this subject, no textbook has previously been published dealing entirely with experimental methods used in determining metallurgical equilibrium diagrams. The monograph now issued is intended to fill this gap.

The theory of the subject is covered by two chapters concerned, respectively, with the general theory of binary and of ternary systems. The remainder of the book deals almost exclusively with experimental methods, including discussion of general procedures, and of methods used for determination of the liquidus, solidus and solid-solubility curves of binary alloys, and the study of ternary systems. In view of the numerous books on X-ray crystallography which are already available, methods for producing X-rays, and the theory of X-ray crystal-structure determination are not treated in detail, but a chapter is devoted to consideration of the precautions necessary when these methods are applied to equilibrium-diagram work. In a final section of the book, the authors make some recommendations on mode of presentation of equilibrium diagrams for publication. The book is intended primarily for Honours and research students in metallurgy.

Three-Stage Electron Microscope in Crystal-Structure Analysis

See abstract on p. 272.

NICKEL

Reduction of Nickel Oxide in a Fluidized Bed

A. KIVNICK and A. N. HIXSON: 'The Reduction of Nickel Oxide in a Fluidized Bed.'
Chemical Engineering Progress, 1952, vol. 48, Aug., pp. 394-400.

Phenomena involved in the 'fluidized bed' have been extensively described during the past few years: the present authors make a condensed classified review of the main literature, supported by a *bibliography* of 30 items. No previous work has been reported in which a reaction taking place in a fluidized bed was affected to any appreciable degree by the rate of gas flow through the bed, i.e., in all research yet reported mass transfer (which would be expected to vary as a function of the gas velocity) did not affect the overall reaction rate, since in each case a kinetic step was the slowest, and therefore the rate-determining, step. The present paper deals with experiments involving the rate of reduction of finely divided solid nickel oxide with hydrogen, in a fluidized bed. The course of the reaction was followed by study of the change in the hydrogen content of the fluidizing gas mixture between the inlet and outlet of the reactor. The results lead to the conclusion that both the diffusional (or mass-transfer) and kinetic steps are of importance in determining the overall reaction rate and that the kinetic step which is effective may be the adsorption of

hydrogen on nickel oxide. It was not found possible to predict the mass-transfer behaviour of the system on the basis of the generalized correlations at present available.

The authors consider that the process might find industrial use as a means of preparing pure nickel from the oxide. The product obtained at the end of one run was found to be 75 per cent. metallic nickel. At that time the bed was still fluidized and no sticking had been observed.

Promotion of Nickel Catalysts

L. D'OR and A. ORZECHOWSKI: 'On Promotion in Heterogeneous Catalysis.' *Comptes Rendus*, 1952, vol. 235, Aug. 4, pp. 368-70.

Study of the promotion of nickel hydrogenation catalysts by ceric oxide.

See also

'Complementary Researches on Promotion in Heterogeneous Catalysis.' *ibid.*, Aug. 11, pp. 430-2.

It is concluded that the efficacy of the promoter addition lies in an increase, so produced, not only in the total surface of the catalyst, but also in the metallic surface, per unit of mass, available for the reaction.

Nickel Kieselguhr

R. B. ROOF: 'The Crystallography of Nickel Kieselguhr.' *Jnl. Chemical Physics*, 1952, vol. 20, July, pp. 1181-2.

Short note reporting investigation of a nickel-kieselguhr substance used as catalyst in organic hydrogenation reactions. Nickel kieselguhr is a diatomaceous earth upon which, and in which, nickel has been chemically deposited. The investigation showed that both nickel and NiO are present and that there is no significant change in lattice parameter in either of these substances. NiO is present in amounts sufficient to give powder lines of appreciable intensity. The authors discuss their observations in relation to those of some earlier investigators, whose conclusions have been somewhat different from those drawn from the experiments now reported.

Recovery of Nickel from Silver-Refinery Waste Liquors

R. K. DUTTA and T. BANERJEE: 'Recovery of Nickel and Zinc from Silver Refinery Waste Liquor: Part II. Chemical Methods.' *Jnl. Scientific and Industrial Research*, India, 1952, vol. 11, Aug., pp. 342-3.

An electrolytic process for recovery of nickel from waste effluent from silver refineries was described earlier by the same authors (*ibid.*, pp. 76-8: *Nickel Bulletin*, 1952, vol. 25, No. 5, p. 128). An alternative chemical procedure, developed later, is described in the second report.

Various methods were tried, based on the solubility of zinc oxide in acids, but none gave satisfactory results, which is to be attributed to the isomorphic character of the oxides of the two metals. Attempts were therefore made to oxidize nickel to a higher-valency state, and then to study the solubility of the oxides in acids and alkalis.

The method developed involves addition of the neutral solution of the waste liquid, with vigorous stirring, to a mixture of caustic soda and sodium hypochlorite, with the result that zinc oxide goes into solution and nickel oxide is precipitated. The precipitate is separated, washed with caustic soda solution and water, dried, and ignited at 500°C. The precipitate was found to contain about 5 per cent. of zinc, even when the treatment was repeated. Further concentration of nickel in the residue, by partial precipitation of nickel as sulphide, or by differential precipitation of nickel and zinc hydroxides at different pH values, was not found possible. Full details of procedure are given.

Determination of Small Amounts of Magnesium in Nickel

S. KITAHARA: 'A Method of Determining a Small Quantity of Metallic Magnesium in Nickel.' *Jnl. Scientific Research Inst.*, Tokyo, 1951, vol. 45, pp. 199-203; *Metallurgical Abstracts*, 1952, vol. 20, Sept., p. 39.

The magnesium content of nickel used in making cathode sleeves of vacuum tubes cannot be determined by reaction with ferric chloride, for reasons which are stated. The following method has been found satisfactory: 'A tube of the material is heated electrically in vacuo by a current of about 13 amp. Most of the Mg evaporates, together with some Ni, leaving MgO behind. Ni is separated from Mg by dissolving in HCl, diluting the solution to 50 c.c., adding 4 c.c. acetic acid (1:1) and 0·5 g. Na acetate, passing H_2S into the boiling solution, filtering, and washing with a solution of H_2S. Filtrate and washings are evaporated to about 20 c.c., allowed to stand overnight after addition of 0·5 g. NH_4Cl and 4 c.c. 10% $(NH_4)_2HPO_4$, and the precipitate is weighed.'

Chromatographic Determination of Nickel

A. LACOURT *et al.*: 'Paper Chromatographic Behaviour of Copper, Nickel and Cobalt; Qualitative and Quantitative Separations.' *Bull. Soc. Chimique de France*, 1951, pp. 873-80; *Chemical Abstracts*, 1952, vol. 46, July 25, p. 6544.

'Details are given for qualitative and quantitative paper chromatographic separations of nickel from copper and of nickel from cobalt, in γ quantities. New solvents and techniques are described. Nickel and copper can be determined by depositing on paper, developing with Me_2CO containing 10 per cent. $CHCl_3$ or dioxane with 10 per cent. AcOH, eluting with HCl, and determining the nickel with dimethylglyoxime and the copper with diethyldithiocarbamate.'

Iodometric Titration of Nickel with Thiosulphate

E. S. TOMULA, O. JUUTINEN and P. TANSKANEN: 'Iodometric Titration of Nickel with Thiosulphate.'

Zeitsch. anal. Chemie, 1952, vol. 135, pp. 265-9; *Chemical Abstracts*, 1952, vol. 46, July 25, p. 6545.

ELECTRODEPOSITION AND OTHER COATING METHODS

Properties of Electrodeposited Nickel: A.E.S. Report

A. BRENNER, V. ZENTNER and C. W. JENNINGS: 'Physical Properties of Electrodeposited Metals. I. Nickel. 3. The Effect of Plating Variables on the Structure and Properties of Electrodeposited Nickel.'
Plating, 1952, vol. 39, Aug., pp. 865-94, 899-927, 933.

This very comprehensive report is issued under the aegis of the American Electroplaters' Research Project No. 9. It incorporates results of co-operative research work at the National Bureau of Standards, where various Divisions assisted in production of deposits, measurement of properties, and collection and correlation of data. The report forms a reference manual of information available from all sources, supported by a *bibliography* of 152 references. (Earlier reports associated with the same Project have appeared in *Proc. Amer. Electroplaters' Soc.*, 1948, vol. 35.)

The main objective of the research has been to accumulate information on those mechanical properties of nickel electrodeposits which are of immediate importance in relation to their engineering applications: a further aim was collection of data on other properties (e.g., density and electrical characteristics, and magnetic and thermal properties) which have significance in determining the suitability of electrodeposits of nickel for special applications. An additional practical purpose of the investigation was the correlation of the physical and mechanical properties of electrodeposited nickel with conditions of deposition, structure, and chemical composition, and the demonstration of the relationships existing among the respective properties.

The nature and mode of presentation of the highly detailed and extensive data recorded are indicated in the skeleton contents-table given below. The general conclusion drawn from the experiments recorded and from the literature is that 'the most important factor that determines the properties of the deposits is the type of solution. Variations in the operating conditions have relatively small effects, provided the conditions are within the satisfactory operating range for the solution used. Effects of bath composition and operating variables, upon properties, as observed in this study, are in essential agreement with data in the literature'. (Minor disagreements are noted.) . .'For the purpose of classification, the nickel baths may be divided into three main groups, according to the magnitude of the physical and mechanical properties of the deposits: (1) the Watts-type solutions, (2) the chloride-type baths, and (3) the bright-nickel baths. No sharp line of demarcation exists between the properties of deposits from these groups: some of the properties of a given deposit may fall in one group and some in another.'

Contents of Report

I. *Introduction:* scope of research; tables showing variation of properties as a function of solution and conditions of deposition.

II. *Preparation of Plating Solutions and Deposits:* compositions; purification methods; addition agents; notes on sheet and tubular test specimens.

III. *Composition of Deposits:* cobalt content; hydrogen and oxygen contents and their relation to operating conditions; chloride content; sulphate content; carbon and sulphur contents.

IV. *Structure of Deposits:* structure observed by optical microscopy, with notes on influence of operating conditions; structure observed by other methods (X-ray diffraction, electron microscopy).

V. *Measurements Related to the Plating Process:* cathode efficiency; internal stress and factors affecting it.

VI. *Density:* method of determination; uniformity of values observed.

VII. *Mechanical Properties:* general observations; methods of measurement (tensile strength, elongation, and Young's modulus, hardness), and reproducibility of measurements; relation between hardness, tensile strength and elongation; relation between mechanical properties and conditions of deposition (including composition of solution); relation between mechanical properties and composition of deposits; Young's modulus of elasticity; fatigue limit; yield strength; internal friction.

VIII. *Electrical and Magnetic Properties:* electrical resistivity (method of measurement, influence of composition of solution and operating conditions; relation between resistivity and other properties); magnetic properties (method of measurement, effect of conditions of deposition, correlation of magnetic and other properties).

IX. *Thermal Properties:* thermal expansivity (mode of measurement and data on individual deposits); specific heat and heat of solution.

X. *Heat-Treatment of Nickel Deposits:* effect on structure and on the various physical and mechanical properties referred to in the preceding sections.

The main practical conclusions are summarized (*vide* col. 1) and a theoretical explanation is advanced to account for the magnitude and wide range of values observed in the physical and mechanical properties of electrodeposited nickel. An appendix to the report contains details of composition and purity of materials used in the N.B.S. experiments.

Zinc in Nickel-Plating Solutions: Effects and Removal

D. T. EWING, *et al.*: 'Effect of Impurities and Purification of Electroplating Solutions. I. Nickel Solutions. 5. The Effects and Removal of Zinc.'
Plating, 1952, vol. 39, Sept., pp. 1033-7.

The paper is one in the series reporting the work of A.E.S. Research Project No. 5, which is concerned with investigation of the effect of traces of metallic impurities in nickel solutions, on the properties of the electrodeposited metal. An earlier report dealt with copper (*ibid.*, 1950, vol. 37, pp. 1157-60: *Nickel Bulletin*, 1950, vol. 23, No. 12, p. 222).

In the experiments now reported, zinc was introduced into the solutions in the form of $ZnSO_4 . 7H_2O$, to give the following concentrations of the metallic element: 2·5, 10, 25, 75, 150 and 300 mg./L. Changes in properties of the deposits were correlated with zinc content, contamination being estimated, on a relative basis, by examination of appearance of the deposits, adherence and ductility tests, evaluation of throwing power, salt-spray tests, and determination of hardness.

Four different baths were used as base: the Watts type at pH 2·2 and 5·2, a nickel-cobalt alloy type at pH 3·75, and an organic type at pH 3·2.

Removal of zinc from nickel-plating solutions was also studied, using two methods: (1) low-current-density electrolysis, and (2) high-pH precipitation, using nickel carbonate.

Conclusions

'No change in the appearance of the coatings from

the Watts pH 2·2 solution occurred until a zinc concentration of 300 mg./L. was reached. At this point there appeared to be a tendency toward brightening of the electroplate. A general overall darkening of the Watts pH 5·2 deposits resulted from zinc concentrations in the range of 10 to 300 mg./L. The presence of zinc in the alloy-type bright nickel solutions resulted in a general decrease in the brightness of the deposits. A concentration of 150 to 300 mg./L. of zinc enhanced the brightness of organic-type coatings. Deposits from all solutions exhibited a darkening effect in the very-low-current-density areas (below 10 amp./sq. ft.: 1·1 amp./sq. dm.), beginning at zinc concentrations of 10 mg./L.

'Concentrations of zinc in the range of 0 to 300 mg./L. caused no change in the adherence of the deposits to the steel base metal.

'An increase in the zinc concentration of the Watts-type solutions resulted in a general decrease in the ductility of the plated layers. No appreciable change in the ductility of the bright nickels was effected by zinc concentrations of 0 to 150 mg./L. At 300 mg./L. the nickel-cobalt type coatings were extremely brittle and highly stressed.

'A slight decrease in the throwing power of the Watts pH 2·2 solution resulted from the presence of 10 to 300 mg./L. of zinc. The other three nickel solutions showed a general increase in throwing power within the range of 0 to 300 mg./L. of zinc. In each case no definite trend was evident.

'An increase in the zinc content of all solutions resulted in a general increase in the corrosion-resistance of the deposits. The organic and nickel-cobalt alloy type electroplated films exhibited a larger increase in resistance to corrosion than did the Watts-type deposits.

'Zinc concentrations of 10 to 300 mg./L. generally caused an increase in the hardness of the nickel plated from the Watts-type (pH 2·2 and 5·2) and organic-type solutions. Certain concentrations of the impurity resulted in a slight decrease in the hardness of the nickel from the alloy-type solution.

'Zinc can be removed by electrolysis from nickel-plating solutions most economically at optimum current densities of 2 to 4 amp./sq. ft. (0·22 to 0·43 amp./sq. dm.). This method permits removal of the impurity in solution to below a concentration of 1 mg./L.

'Concentrations of zinc below 250 mg./L. cannot be removed from nickel solutions by the high-pH precipitation method without appreciable loss of nickel. The method is, therefore, not recommended for the removal of zinc from nickel-plating solutions.'

Cast Nickel Anodes

E. R. THEWS: 'The Production of Cast Nickel Anodes.' *Metal Finishing*, 1952, vol. 50, Sept., pp. 70-4, 77.

A general review, including discussion of the composition and degree of purity requisite in cast anodes, the advantages of cast anodes (mainly high solubility), and their limitations, chief of which is anode-sludge formation. The object of the article is to describe production methods which may be used to obviate disadvantages sometimes arising from the use of this type of anode.

In order to secure best results it is considered that the following requirements must be satisfied:—

1. The nickel must be as pure as possible, and melting and casting processes must be adjusted to produce dense, gas-free castings.
2. Cooling of the castings must be effected in such a way as to produce a uniform and fine-grained structure.
3. The surface of the castings must be absolutely clean, and free from casting skin.

Detailed recommendations are made on selection of raw materials, melting and casting, and cleaning.

'Udytal' Process of Direct Nickel Plating of Aluminium

J. BARY: 'The 'Udytal' Process of Direct Nickel Plating.' *Revue de l'Aluminium*, 1952, vol. 29, No. 184, pp. 16-17.

The initial layer of nickel is deposited, on a zincate-treated surface, from a special bath (composition not stated), which is capable of tolerating some degree of contamination with zinc without impairment of the properties of the nickel plating. Bright-nickel plating is then deposited (from a Udylite solution), and a final chromium coating is made from a bath containing chromic acid 300 g., sulphuric acid 3 g., per litre. It is claimed that the Udytal process is applicable to all types of aluminium alloy, cast and wrought, and that omission of the copper-plating step, and reduction in the number of buffing operations required, result in substantial saving of time and cost.

Costing and Control of Materials in Nickel Plating

R. T. HOOD: 'Material Control in Nickel Chrome Plating.' *Metal Finishing*, 1952, vol. 50, Sept., pp. 64-6.

The writer gives details of methods of recording consumption of materials and costs in plating operations, from which estimates for work and standard costs may be made with a high degree of accuracy. Typical schedules are tabulated.

Silver Plating of Nickel-Iron Alloys

R. T. FOLEY, R. D. ALFORD and J. K. EASLEY: 'The Electrodeposition of Silver on an Iron-Nickel Alloy for a Glass-to-Metal Seal Arrangement.' *Jnl. Electrochemical Soc.*, 1952, vol. 99, Sept., pp. 349-53.

In the manufacture of cast glass bushings for electrical apparatus lead-free borosilicate glass, in the molten state, is poured into a mould containing an iron-nickel alloy flange. After cooling slightly, the cast structure is moved to a furnace at about 550°C., annealed for several hours, and finally removed from the furnace at about 100°C. Although iron-nickel alloys possess considerable resistance to high-temperature oxidation, scaling occurs during this process, and the scale formed must be removed before the flange can be soldered during assembly of the apparatus. It is therefore of importance to protect the alloy from oxidation during processing, and one means of conferring such protection is by electrodeposition of

silver. Considerable work has been done on electrodeposition of silver on mild steel, but it was considered improbable that this could be directly applied to plating of iron-nickel alloy, and it is with the deposition of silver on a 58-42 per cent. alloy that this paper specifically deals. The thermal-expansion curve of this material matches quite closely that of a lead-free borosilicate glass. As a preliminary step, the alloy is annealed at 1125°C. in a hydrogen atmosphere (dew point about −50°F.), a treatment which is effective in degassing and decarburizing the metal (methane reaction), so that no bubbling takes place when the glass is cast.

In order to develop the properties required for this particular application (a surface which would participate in the glass-to-metal seal, and also be resistant to diffusion of oxygen), it was found necessary to cover the silver plate with a thin coating of indium. The process therefore involved surface preparation of the iron-nickel alloy, silver 'striking', silver plating, and indium plating. Detailed discussion of conventional operations such as degreasing and rinsing is omitted, but the other processes are described in detail. Before the initial pickling the samples were vapour-degreased in trichlorethylene, and between successive operations, with the exception of the silver-striking and -plating steps, the parts were rinsed in warm water.

The initial hydrogen firing at 1125°C. produced an oxide layer which was difficult to remove by conventional procedures. Two methods of pickling were tried: the one recommended involves cathodic treatment at room temperature in one or other of the following solutions: (1) 1 part by volume of concentrated hydrochloric acid (37%), 1 part of concentrated nitric acid (70%) and 9 parts of water, or (2) a 10% solution of sulphuric acid. A cathode current density of about 2·6 amp./sq. dm. is used, for 15 minutes. Carbon anodes gave the best results.

The silver 'strike' bath contained 1·0 g./L. of silver cyanide and 80 g./L. of potassium cyanide. Stainless steel anodes were used. An essential factor is that the part to be plated be carried from the neutralizing step to the 'strike' bath without drying, and that it be immersed in the bath with the current on. Treatment is with a current density of about 1·7 amp./sq. dm., usually for about 30 secs. The silver-plating solution contains silver cyanide 30 g./L., potassium cyanide 42 g./L., potassium carbonate 45 g./L. Current density is 0·4 amp./sq. dm., and operation is at room temperature. Silver anodes are used.

After silver plating, the parts are fired in hydrogen for 15 minutes at 850°C., which constitutes an adhesion test, since it has been found that parts which show no blistering after this treatment go satisfactorily through the manufacturing process, which includes the casting operation and the annealing in air (see p. 259). There is also evidence that such a firing operation improves adherence.

The silver-plated parts, after annealing, are dipped into a solution of potassium cyanide (50 g./L.), and are then indium-plated in a conventional cyanide bath. Subsequent annealing treatment for diffusion of the silver and indium is found to be unnecessary, since during normal operation the part is at elevated temperature, and considerable diffusion undoubtedly occurs in service.

The paper contains informative illustrations showing the nature of the interfaces formed at various stages of the process.

Adhesion Testing of Electrodeposited Metals: Modified 'Nodule' Method

H. C. SCHLAUPITZ and W. D. ROBERTSON: 'Quantitative Measurement of Adhesion of Electrodeposited Metals.' *Plating*, 1952, vol. 39, July and Aug., pp. 750-3, 764; 862-4, 932.

The general problem of evaluating adhesion of electrodeposits is considered to be analogous to that of measuring the fracture strength of metals in the absence of appreciable plastic deformation. Features requisite in an ideal adhesion test are considered: the test specimen must incorporate (1) symmetry across the interface in question, (2) absence of local stress concentration, (3) a minimum thickness of base metal and deposit, to limit plastic deformation, (4) a means for transmitting the load to the interface which subjects both base metal and deposit to a stress state of high triaxial tension. Mechanically, certain essentials with regard to method of gripping must also be satisfied. Methods which have previously been proposed for adhesion testing (Burgess method, Ollard method and modifications of it, shear-test method, and Brenner 'nodule' method) are evaluated on the basis of these ideal requirements, and it is concluded that none of the procedures is entirely satisfactory and reliable.

The improved procedure devised by the present authors is based on the Brenner 'nodule' method, modifications being introduced to eliminate high stress concentration at the base of the nodule, and to provide symmetry across the interface. A sketch of the specimen, photographs of the grips used, and details of operation of the test are given in the paper. Using this procedure, it is found possible to cause non-ductile fracture in annealed copper foil when the design of the test specimen is such that a triaxial state of tensile stress is obtained.

NON-FERROUS ALLOYS

Induction-Furnace Melting of Nickel and Nickel Alloys

B. SPINDLER: 'Induction Furnace Melting of Corrosion-Resistant Alloys.' *Foundry*, 1952, vol. 80, Aug., pp. 110-13.

The article describes practice at the foundry of Alloy Steel Products Company, Linden, N.J., in production of nickel-chromium-iron, nickel, Monel, Inconel, and nickel-molybdenum-iron castings. The charges consist in most cases of 60 per cent. scrap from previous heats, with 40 per cent. steel, ferroalloys, or special ingots. Furnace linings are basic, being made up by ramming dry Normagal between a crucible-shaped transite inner form and the furnace coil, which has been previously protected by an air-setting plastic refractory cement. The inner form remains in place during the

melt-down, and holds its shape sufficiently long to permit the granular refractory to sinter to a depth which will render it self-supporting. Moulds are made according to standard green-sand practice, using synthetic sand for the moulds and oil sand for the cores. Most of the moulds are hand-rammed, with pneumatic rammers. For transfer of the molten metal from the furnace to the moulds teapot ladles are used for the smaller pieces, and standard lip-pour ladles for the larger items. The full melting cycle for 600/670-lb. heats averages about one hour and ten minutes, of which only five minutes is required for pouring, the rest being for melting and superheating. Charging time is practically negligible. It is found that the high speed of the melting cycle permits realization of an output far in excess of the normal furnace capacity. The cooled castings are shaken out of the moulds, and blast-cleaned; gates and risers are cut off, and surfaces are rough-ground. Most of the castings are heat-treated at 2050°F. (1120°C.), for one hour per inch of section, and are water-quenched. For some of the complex nickel-base alloys higher temperatures, and air-cooling, are used. After heat-treatment the castings are again blast-cleaned, pickled, and inspected. The layout of the casting area is illustrated, and a diagrammatic description is given of the abrasive cutting-off wheel used for removing gates and risers from large castings.

Influence of Grain Size on High-Temperature Properties of Monel

See abstract on p. 268.

Creep-Rupture and Recrystallization Studies on Monel

See abstract on p. 268.

Low-Copper Nickel-Copper-Zinc Casting Alloy

LOS ALAMOS SCIENTIFIC LABORATORY, UNIVERSITY OF CALIFORNIA: 'Low-Copper Nickel Silver of Improved Chemical and Physical Properties.' *Report LA* 1324, Nov. 1, 1951; 34 pp.

Report of work done under contract for the U.S. Atomic Energy Commission.

The investigators are of the opinion that the commercial potentialities of low-copper, high-nickel, high-zinc nickel silvers have in the past been overlooked, due, in part, to the difficulty of preventing high zinc losses in melting. The slag cover described in this report obviates excessive loss, and makes it possible to control composition of the melted product within ±0·75 per cent. for all constituents. The alloy discussed contains copper 40, nickel 32, zinc 28, per cent.: it proved to be a one-phase solid solution, and, under suitable conditions of melting, gave dense, gas-free castings.

The following notes are given with regard to production and properties.

Melting and Casting Procedure

Alloy charges weighing from 15 to 300 lb. were melted in induction furnaces provided with adequate hooding to remove zinc fumes. All melts were made in commercial clay graphite crucibles. Accurate determination of temperature was impractical, due to the heavy evolution of zinc fumes near the pouring temperature, but temperature control was accomplished by means of a Chromel-Alumel direct-reading, protected-tip, lance pyrometer.

For each charge (comprising balanced proportions of commercial nickel silver, brass, nickel-copper alloys, nickel, and zinc) an addition of manganese dioxide equivalent to 0·4 per cent. of the total charge was placed in the bottom of the crucible, to provide a mildly oxidizing condition. On reaching the refining temperature the melts were deoxidized with phosphorus, manganese and magnesium. The slag cover used contained soda ash 52, borax 27, silica 21, per cent. (m.p. about 815°C.): it proved highly effective in minimizing loss of zinc without impairing the quality of the cast metal. Prior to pouring the metal the slag was thickened with silica sand, which coagulated the slag, thus permitting removal by skimming.

At the maximum temperature used during melting (1175°C., at the refining and deoxidation stage) evolution of zinc-oxide fumes was vigorous, in spite of the slag cover, and the narrow range between boiling point and liquidus made it necessary for the metal to be poured as soon as possible after deoxidation and cutting of the power from the furnace.

Moulds

Castings produced in graphite moulds were dense and free from internal defects, but the surfaces were rough and irregular, due, it is believed, to volatilization of the zinc in the mould. The surface defects were eliminated by the use of moulds having a silica-sand base, with additions of silica flour, bentonite, core oil, cereal flour and water. The moulds were dried overnight at 170°C.

Mechanical Properties of the Castings

Mechanical tests were made on specimens of the cast alloy, in comparison with samples of nickel silver of the copper 53·5, nickel 20, zinc 26·5, per cent. type. The high-nickel alloy showed 50 per cent. higher tensile strength, 30 per cent. higher yield strength (0·2 per cent. offset), 20 per cent. higher modulus of elasticity, and 40 per cent. lower elongation than the lower-nickel alloy.* The hardness of the former was also considerably higher than that of the more conventional composition.

Corrosion Tests

Samples of both types of material were exposed in aqueous solutions containing acetic acid (10% by wt.) +3% sodium chloride, and in 10 per cent. solutions of ammonium sulphate, calcium hypochlorite, cupric nitrate, ferrous chloride, hydrochloric acid, nickelous chloride, potassium hydroxide, zinc chloride, and trisodium phosphate. All tests were made at room temperature and in non-aerated solutions. The general conclusions drawn were as follows:

(*a*) The low-copper alloy had superior corrosion resistance in six of the media tested: acetic

* The 20 per cent. nickel silver contained less tin, lead and iron than the usual commerial alloy of this type, which is thought to account for the low strength and high ductility observed.

acid and sodium chloride, ammonium sulphate, cupric nitrate, ferrous chloride, nickelous chloride and zinc chloride.

(*b*) Corrosion in calcium hypochlorite solution was initially great on both metals, and corrosion of both alloys was roughly the same for the extent of the tests.

(*c*) Essentially no corrosion of either sample was detected in tri-sodium phosphate or potassium hydroxide solutions.

(*d*) In hydrochloric acid the low-copper alloy had inferior corrosion-resistance for the first part of the tests and then superior resistance for the remainder of the time.

It is considered that although the casting properties of the high-nickel, low-copper alloy were suitable for the shapes immediately required, minor additions of tin would still further improve castability, without adversely affecting physical or chemical properties. The undesirably high solidification shrinkage observed could be minimized by modified design, and the extent of the shrinkage could be reduced, with some loss of casting density, by less severe deoxidation procedure. Since the crystal structure of the modified alloy is face-centred cubic, the material should be amenable to working to wrought products, by techniques similar to those used for other grades of nickel silver. The properties of the alloy indicate its possible economic use for corrosion-resisting springs, precision instruments, and pressure applications, and in the marine, architectural, and food-handling industries.

Nickel-Alloy Thermocouples: British Standard

See abstract on p. 256.

High-Nickel Alloys used in Aircraft

HENRY WIGGIN AND CO., LTD.: 'High-Nickel Alloys in Aircraft Construction.' *Publn.* 613, 1952; 20 pp.

A condensed review of some major applications of high-nickel alloys in aircraft, demonstrating the reasons which have dictated selection of the respective materials (on the basis of service requirements) for the various components described.

Nickel-containing materials are discussed in relation to their use in gas-turbine parts; piston engines; compound engines; accessories and instruments; fuel and oil systems; fastenings, rivets and bolts. The materials covered by the publication are the Nimonic series, Inconel, Monel, K Monel, nickel, and electrical-resistance materials.

Centrifugal Castings of Nickel-Brass and -Bronze: A.S.T.M. Specification

AMER. SOC. TESTING MATERIALS: 'Proposed Tentative Specification for Copper-base Alloy Centrifugal Castings.' Contained in Report of Committee B-5 on Copper and Copper Alloys.
A.S.T.M. Preprint 11, June, 1952, pp. 17-18.

The specification covers centrifugal castings of alloys to a number of A.S.T.M. specifications, including classes 10A (leaded nickel brass) and 11A and 11B (leaded nickel bronze) referred to in schedule B 149.

Hall Effect in Cobalt-Nickel Alloys

E. M. PUGH and S. FONER: 'Ordinary Hall Effect in Cobalt-Nickel Alloys.' *Physical Rev.*, 1952, vol. 87, July 1, p. 210 (Abs. N.6).
Abs. of paper to Washington Meeting of the American Physical Society, May 1952.

'Measurements at room temperatures of the Hall effect in the cobalt-nickel series of alloys have been made. The ordinary Hall coefficient is negative (as in the nickel-copper series) throughout the range. The values agree, within a factor of 2, with values predicted from the assumption that from 0·6 to 0·7 *s* electrons per atom contribute to the conduction.

'This result is similar to that found by SCHINDLER and PUGH in the ferromagnetic alloys of copper-nickel. Except in the immediate region of pure nickel, the ordinary coefficient in these ferromagnetic alloys is larger than predicted, so that the discrepancy cannot be explained by assuming any *d* band conduction.'

Elastic Properties of Nickel-containing Aluminium Alloys

N. DUDZINSKI: 'The Young's Modulus, Poisson's Ratio and Rigidity Modulus of Some Aluminium Alloys.' *Jnl. Inst. Metals*, 1952-53, vol. 81, pp. 49-55; *Monthly Jnl.*, Sept., 1952.

Determinations on binary, ternary and quaternary alloys are reported. The binary and ternary series studied included alloys in which nickel was present in various amounts.

Nickel-Rich Nickel-Titanium-Aluminium Alloys

A. TAYLOR and R. W. FLOYD: 'The Constitution of Nickel-Rich Alloys of the Nickel-Titanium-Aluminium System.' *Jnl. Inst. Metals*, 1952-53, vol. 81, pp. 25-32; *Monthly Jnl.*, Sept., 1952.

The investigation reported was undertaken as part of a study, by micro-examination and X-ray analysis, of the phase relationships in alloys containing more than 50-at. per cent. nickel, over the temperature range 750°-1150°C. Recent, closely associated, work on the nickel-titanium binary system was described *ibid.*, 1951-52, vol. 80, pp. 577-87; *Nickel Bulletin*, 1952, vol. 25, Nos. 8-9, p. 202.

The present paper includes results of a study of the nickel end of the nickel-aluminium phase diagram, carried out to clear up discrepancies among the several diagrams published previously, none of which could be satisfactorily linked with data for the ternary system. A complete new phase diagram, incorporating the new boundaries defined by this work, and the latest data available in the literature, is given. *Inter alia*, this diagram includes revision of the γ and γ' phase fields. For details of the nickel-titanium system, reference is directed to the earlier paper (*loc. cit.*): the tentative nickel-titanium phase diagram developed as a result of the authors' work is reproduced in this paper.

The main findings with regard to the ternary system, following the extensive micrographic and X-ray studies made in the present research, are as follows:—

At 750°C. the face-centred cubic primary nickel solid

solution, γ, holds more than 12 at.-% titanium and aluminium in solution. The γ phase is in equilibrium with the intermetallic compound η-Ni_3Ti and the extensive face-centred cubic ordered phase, γ', based on Ni_3Al. A brief survey of alloys with 50 at.-% nickel has revealed a ternary Ni_2TiAl phase, β_3, with a Cu_2ZnAl type of structure, in equilibrium with the β_1-NiAl and β_2-NiTi phases, which have CsCl-type structures.

Metallurgical Equilibrium Diagrams: Textbook

See abstract on p. 256.

NICKEL-IRON ALLOYS

Influence of Temperature on Eddy-Current Loss in Thin Nickel-Iron Alloy Tape

C. ABGRALL and I. EPELBOIN: 'Investigation of the Temperature Variations of Thin Mumetal Tapes subjected to Weak Alternating Fields.' *Comptes Rendus*, 1952, vol. 234, Mar. 17, pp. 1265-7. *Wireless Engineer*, 1952, vol. 29, Sept., p. A. 187, Abs. 2529.

'The calculated value of eddy-current losses for a homogeneous magnetic material accounts for only a part of the experimentally observed losses. The variation of the discrepancy over a wide temperature range was investigated; this enables the influence of magnetic after-effect to be separated from that of macroscopic structure. Experimental results are given for tapes of thickness 10μ and 59μ.'

Nickel-Iron-Molybdenum Ferromagnetic Alloy (Vibralloy)

M. E. FINE: 'Vibralloy: A New Ferromagnetic Alloy.' *Bell Laboratories Record*, 1952, vol. 30, Sept., pp. 345-8.

During the past few years the metals research group of the Bell Telephone Laboratories has been studying the fundamental factors controlling the modulus of elasticity of metals, and its variation with temperature. This paper describes an alloy containing about 40 per cent. of nickel and 9 per cent. of molybdenum, balance iron, in which, by suitable processing and heat-treatment, it is possible to develop an elastic modulus which can be controlled over a relatively wide range of temperature, substantial ferromagnetic permeability, and high mechanical strength, a combination of properties particularly attractive in material for reeds of vibrating reed selectors. In such selectors, tuned reeds vibrate when driven magnetically by a signal of the proper frequency. These reeds must have strength adequate for mechanical stability, and they must respond to the same signal frequency over a relatively wide temperature range, approximately $-40°$ to $+80°$C. Since the reed is magnetically actuated, moreover, it must have substantial ferromagnetic permeability over the full temperature range. The tuned frequency of a reed, that is the signal frequency to which it responds, depends upon its dimensions, on the density of the reed material, on Young's modulus of the material, and on the parameters of the magnetic circuit of which it is a part. In general, all these factors change with temperature, and the response frequency of the reed will also change with temperature unless the changes in frequency due to the four factors are balanced. The inter-relationship of ferromagnetic properties and elastic modulus in iron-nickel alloys is discussed and attention is directed to compositions in which constancy of elastic modulus has been secured over a certain temperature interval, as in Elinvar.

Development of these alloys has been taken a step farther by evolution of the Vibralloy type, in which cold-working plays an important part in widening the range of constancy of modulus and in increasing hardness and mechanical strength. After cold-working, the alloys are annealed at 400°-500°C. Vibralloy shows a substantially constant modulus of elasticity over the range $-40°$ to $+80°$C.

The molybdenum addition in these alloys is made with a dual purpose: (1) it increases mechanical strength (the molybdenum-containing alloy has a much higher limit of elasticity than the molybdenum-free type, and is consequently a better spring material), and (2) thermal variation of modulus in the Vibralloy composition is less sensitive to changes in nickel content than in the molybdenum-free alloys. Molybdenum reduces the ferromagnetism of the alloy to some degree, but the magnetic properties are still adequate for efficient magnetic actuation of vibration.

Curves given in the paper show temperature variations in elastic modulus of cold-worked nickel-iron alloys of various nickel contents (40·8-45·0), and illustrate also the influence of molybdenum in reducing the sensitivity of the mean thermal coefficient of modulus to changes in nickel content.

Ultrathin Nickel-Iron Alloy Tape: Properties and Uses

M. F. LITTMAN: 'Ultrathin Magnetic Alloy Tapes with Rectangular Hysteresis Loops.' *Electrical Engineering*, 1952, vol. 71, Sept., pp. 792-5.

The continuing need for magnetic core materials which can be effectively used at high frequencies has stimulated the search for materials in which eddy currents produced by rapid changes in magnetic flux are at a minimum. Generally speaking, there are two ways in which eddy-current effects can be reduced: (1) increase in electrical resistivity (as in the ferrites), or (2) restriction of the paths in which eddy currents can flow, as by use of powdered metals or thin laminations of metal strip. Use of ferrites is limited in certain applications by their relatively low magnetic saturation, low Curie point and certain dielectric properties. In powder-metal cores the fine sub-division of the particles results in a very low d.c. permeability, even for the best alloys. Where high permeability is essential, very thin tapes are useful, and it is with cores made from ultrathin tape that this article is concerned.

The commercial use of magnetic alloys rolled to thin gauges grew rapidly during the war period; for power

transformers, laminated or tape-wound cores of strip 4-6 mils thick were employed, and for radar pulse transformers cores of 1-3 mils thick material were used. Already during that period, however, there were requirements for rates of magnetization so high that materials even thinner than 1 mil (0·025 mm.) were required, and small quantities of 4-79 molybdenum Permalloy $\frac{1}{4}$ mil thick were rolled on a small mill. Since 1950 four different alloys have been rolled to gauges of less than 1 mil and the tape has been fabricated into toroidal cores. Some values characteristic of these alloys are tabulated below: it is considered that they probably do not change with thickness in the range 1 mil to $\frac{1}{8}$ mil.

Although a detailed picture of the magnetic properties of ultrathin magnetic alloys cannot be presented at this early stage of development, tests made to date (using the ballistic galvanometer) give some general idea of the type of magnetic characteristics which may be expected under d.c. magnetization. The conclusions so far reached are surveyed in this article, which includes diagrams showing the d.c. hysteresis loops of grain-oriented 3 per cent. silicon-iron, grain-oriented 48 per cent. nickel-iron, and 4-79 molybdenum Permalloy, for tapes of varying thicknesses (1 to $\frac{1}{8}$ mil). Comparison is also made of hysteresis loops for four magnetic alloys in ultrathin tape ($\frac{1}{4}$ mil), and a further figure shows variation of ratio of residual to maximum induction with magnetizing force for cores of $\frac{1}{4}$ mil tape of four alloys having rectangular hysteresis loops. In this last figure, data for 1-mil grain-oriented 48 per cent. nickel-iron alloy are included for comparison.

It is considered too early to give a balanced opinion on the potential capabilities of these ultrathin materials under pulse excitation or continuous high-frequency operation. It is believed, however, that the 48 per cent. nickel-iron and the 4-79 molybdenum-Permalloy tapes show considerable promise for use in high-speed memory cores in computers. For magnetic amplifiers and radar pulse transformers, the low coercive force and high magnetic saturation characteristic of the ultrathin tapes are of interest, although it may not necessarily be desirable in such cases to have a rectangular d.c. hysteresis loop. Ultrathin 4-79 molybdenum Permalloy appears to be particularly attractive, since it has a relatively high volume resistivity and can be made to have either a rectangular or 'rounded' d.c. hysteresis loop, together with low coercive force.

Nickel-Alloy Magnetic Cores for Small Power Transformers

A. V. HUGHES and C. F. SALT: 'Magnetic Core Materials for Small Power Transformers.'
Electrical Manufacturing, 1952, vol. 49, June, pp. 133-8, 324-336 (even numbers only).

This paper gives a detailed report of an investigation and evaluation of various configurations of core materials for optimum transformer design, carried out at the Battelle Memorial Institute, under the sponsorship of the U.S. Signal Corps. 'Optimum design' in this case is to be understood as being that design which has minimum size and weight for a given set of performance requirements.

The discussion includes details of the properties and uses of various types of nickel-iron and nickel-cobalt-iron alloys used in such cores, and consideration of the effects of thickness of lamination used, joints, core clamps, temperatures, and frequency of operation, on service performance.

Nickel-Alloy Magnets in Switch Contacts

R. B. IMMEL: 'Permanent Magnets Increase Capacity and Life of Switch Contacts.' *Machine Design*, 1952, vol. 24, Sept., pp. 132-8.

The author deals in detail with the advantages gained by addition of permanent magnets to contacts on direct-current switches. Effective arc blow-out field is thus provided, and interrupting capacity is much increased. In many cases a single switch unit equipped with a permanent-magnet blow-out can handle a circuit normally requiring two standard switch units in series. Typical assemblies incorporating permanent magnets are described and illustrated.

The permanent magnets used are of the Alnico (nickel-aluminium-cobalt-copper-iron) type: methods for selecting the most suitable grade of alloy are discussed, with critical evaluation of the suitability, for this application, of the respective compositions, on the basis of properties, performance and cost. On balance, Alnico II has proved to be the best choice: it is characterized by relatively good field strength, and has satisfactory resistance to demagnetization. Its cost is only slightly higher than that of the least expensive grade of the alloys which could come into consideration for the purpose.

Basic Properties of Alloys Studied

	Silicon 3 % Bal. Iron	Nickel 48 % Bal. Iron	Nickel 79 % Molybdenum 4 % Bal. Iron	Nickel 79 % Molybdenum 5 % Bal. Iron
Magnetic Saturation, gauss	20,000	16,000	8,700	8,000 +
Curie Temperature, °C. ..	700	440	420	400
Resistivity, microhm-cm. ..	47	45	55	60
Density, g./c.c. ..	7·65	8·25	8·72	8·77

Magnetic Viscosity of Alnico under Variable Field Conditions

R. STREET, J. C. WOOLLEY and P. B. SMITH: 'Magnetic Viscosity under Discontinuously and Continuously Variable Field Conditions.' *Proc. Physical Soc.*, Sect. B, 1952, vol. 65, Sept., pp. 679-96.

For a ferromagnetic specimen exhibiting magnetic viscosity, the intensity of magnetization increases continuously with time under steady external-field conditions. This paper gives an account of investigations made to determine the influence, on magnetic viscosity, of changes in the magnetic field applied to the specimen. Two cases of discontinuous and continuous changes in field are considered. The measurements were made on Alnico of the following approximate composition: aluminium 10, nickel 18, cobalt 12, copper 6, iron 54, per cent. Both cases can be adequately described by extending the formal theory of magnetic viscosity previously proposed, in which it was supposed that the domain process responsible for magnetization can be activated by thermal agitation.

The required extension of the theory consists in assuming that the activation energy of the domains is a function of the field. The experimental results show that the changes in domain activation energy are linearly proportional to the changes in field strength, if the latter are small. Denoting the constant of proportionality by q, and considering a possible domain model, it is shown that q is related to the elementary volume of the material in which thermal agitation leads to activation. Measurements on the Alnico specimens used show that the linear dimensions of this elementary volume are of the same magnitude as the width of a domain boundary wall. Results of the variation of q and other parameters characteristic of magnetic viscosity as functions of the temperature of the specimen are also presented and it is shown that the observed behaviour is consistent with the present views on the metallurgical structure of Alnico.

Magnetic Study of Nickel-Iron Alloy having Low Internal Stress

I. EPELBOIN and G. GILARDIN: 'Study, in Static and Alternating Fields, of a Ferro-Nickel having Very Low Internal Stress.' *Comptes Rendus*, 1952, vol. 234, May 5, pp. 1860-2.

Mumetal was studied, with particular reference to phenomena observed in very thin strip. Magnetic texture is shown to be associated with a surface demagnetizing effect, which would explain why very thin strip is characterized by relatively low permeability, whatever the heat-treatment undergone, and however pure the atmosphere in which it is carried out. Comparative data on Mumetal strip of 50-6 mu thick are tabulated, to demonstrate the influence of thickness on magnetic permeability.

Silver Plating of Nickel-Iron Alloys

See abstract on p. 259.

CONSTRUCTIONAL STEELS

Critical Temperatures for Plain and Alloy Steels: Data Sheet

'Approximate Critical Temperatures for Steels.' *Metal Progress*, 1952, vol. 62, Sept., p. 96B; Data Sheet.

Tables compiled by the Laboratory of the Central Alloy District, Republic Steel Corporation. Details of Ac_1 and Ac_3 on slow heating, and of Ar_3 and Ar_1 on slow cooling. In some cases the M_s temperature is also included. Information is tabulated on carbon, free-cutting, manganese, nickel, nickel-chromium, molybdenum, chromium, chromium-vanadium and low-triple-alloy steels.

Influence of Silicon on Tempering of Martensite in Nickel Steels

A. G. ALLTEN and P. PAYSON: 'The Effect of Silicon on the Tempering of Martensite.' *Amer. Soc. Metals, Preprint* 10, Oct., 1952; 28 pp.

In a study of the properties of Hy-Tuf steel (typical composition carbon 0·25, manganese 1·30, silicon 1·50, nickel 1·80, molybdenum 0·40, per cent.) it was found that this steel, when oil-hardened and subsequently tempered, did not soften during tempering in the range 300°-700°F. (150°-370°C.) as did other medium-alloy steels. It was ultimately established that the anomalous behaviour was due to the silicon content of the steel, and, following a review of the literature, the present authors made an investigation of the mechanism by which silicon retards softening of martensite. Most of the work was done on four materials: 0·6 per cent. carbon steels containing 0·4 and 2·2 per cent. silicon, and steels containing carbon 0·4, nickel 3, per cent. with silicon at 0·5 and 2·2 per cent. Data from some other steels are included for confirmation, including results on 3 per cent. nickel steels containing 0·8, 1·5, 3·0 and 3·7 per cent. of silicon. Hardness, dilatometric, specific volume, electrical-resistivity and X-ray diffraction measurements were used in examination of the steels.

The results demonstrate that the temperature at which cementite forms, on heating of the martensite of steels at a definite rate, is raised in relation to the silicon contents of the steels. The temperature at which cementite formed in the 3 per cent. nickel-base steels was raised about 300°F. (170°C.) by an increase of silicon from 0·5 to 2·2 per cent. Epsilon iron carbide was detected in the 0·6 per cent. carbon steels after tempering at lower temperatures than those required to produce cementite.

The softening of the 2·2 per cent. silicon steels was retarded in the tempering range of 400°-600°F. (205°-315°C.). The hardness after a given temper in that range was found to depend only on the silicon and carbon contents of the steels. A mechanism which involves coherency hardening is postulated as the cause of the retarded softening of the high-silicon steels.

The notch impact properties of the tempered steels were affected by the presence of silicon. The transformation of retained austenite was retarded as a result of the presence of silicon in the steels containing 2·2 per cent. Silicon did not precipitate as a compound or in the carbides during the various stages of tempering.

Influence of Various Forms of Heat-Treatment on Temper Brittleness

L. D. JAFFE, D. C. BUFFUM and F. L. CARR: 'The Effect of Various Heat-Treating Cycles upon Temper Brittleness.' *Amer. Soc. Metals, Preprint* 25, Oct., 1952; 7 pp.

Although much work has already been done on the effect of isothermal and of furnace-cool embrittling treatments on the temperature of transition from tough to brittle fracture of steels susceptible to temper-brittleness, there has been little study of the degree of embrittlement (as indicated by transition temperature) developed by other heating and cooling cycles. The work reported in this paper was planned to investigate the effect of various heating, holding, and cooling cycles, in the ferrite range, on the transition temperature of a susceptible steel, S.A.E. 3140. The steel used contained carbon 0·39, manganese 0·79, silicon 0·30, sulphur 0·028, phosphorus 0·015, nickel 1·26, chromium 0·77, molybdenum 0·02, vanadium less than 0·01, nitrogen 0·006, per cent.

Blanks (2 $\frac{3}{16}$ in. long) of bar stock were austenitized at 900°C. for one hour, water-quenched, and tempered for one hour at 675°C. The blanks were then divided into 20 groups of 12 blanks each, and were further heat-treated as shown in the table below, which also summarizes the results.

The observations made show that:—

(1) Quenching to room temperature between tempering and isothermal temper-embrittlement treatments had no significant effect upon the resulting embrittlement.

(2) Slow heating to the isothermal embrittlement temperature of 500°C. introduced the same amount of embrittlement as slow cooling from

Summary of Heat-Treatment and Results

Group	Cooling from Tempering	Embrittling Treatment				Hardness Rockwell, C	Transition Temp., °C.
		Heating Method	Temp. °C.	Time, hours	Cooling		
1	Water-quenched					24	−80
2	17°C/hr. to R.T.*					22	−10
3	Water-quenched	Salt at ..	500	1	Water-quenched	22	−65
4	Water-quenched	Salt at ..	500	1	17°/hr. to R.T.	22½	−55
5	Water-quenched	Salt at ..	500	48	Water-quenched	23½	0
6	Water-quenched	Salt at ..	500	48	17°/hr. to R.T.	23½	+20
7	Water-quenched	17°/hr. to ..	500	1	Water-quenched	24	−65
8	Water-quenched	17°/hr. to ..	500	1	17°/hr. to R.T.	23	−40
9	Water-quenched	17°/hr. to ..	500	48	Water-quenched	23	+15
10	Water-quenched	17°/hr. to ..	500	48	17°/hr. to R.T.	23	+25
11	Salt at 500°C.			0	17°/hr. to R.T.	23	−55
12	Salt at 500°C.		500	1	Water-quenched	22½	−65
13	Salt at 500°C.		500	1	17°/hr. to R.T.	23½	−50
14	Salt at 500°C.		500	48	Water-quenched	24	+ 5
15	Salt at 500°C.		500	48	17°/hr. to R.T.	24	+25
16	17°/hr. to 500°C.			0	Water-quenched	20½	−20
17	17°/hr. to 500°C.		500	1	Water-quenched	22	−25
18	17°/hr. to 500°C.		500	1	17°/hr. to R.T.	21½	− 5
19	17°/hr. to 500°C.		500	48	Water-quenched	22	+20
20	17°/hr. to 500°C.		500	48	17°/hr. to R.T.	22½	+35

*Room Temperature.

this temperature, within experimental error. This conclusion may not apply to other temperatures.

(3) Most of the embrittlement produced by slow cooling from the tempering temperature of 675°C. developed above 500°C.

(4) For the heat investigated, embrittlement developed more rapidly in treatments involving continuous cooling than during isothermal holding at any temperature.

(5) When several embrittlement treatments were applied successively to the same specimens, each contributed to the embrittlement. When the transition temperature was high, the introduction of an additional embrittlement treatment often did not produce as large an increase in transition temperature as when the prior transition temperature was low.

Effect of Hardness on Impact Energy in Temper-Brittle and Unembrittled Steel

F. L. CARR, M. GOLDMAN, L. D. JAFFE and D. C. BUFFUM: 'Effect of Hardness on the Level of the Impact Energy Curve for Temper-Brittle and Unembrittled Steel.' *Amer. Soc. Metals, Preprint* 26, Oct., 1952; 5 pp.

During work carried out by some of the authors on transition temperatures (*Trans. Amer. Inst. Mining and Metallurgical Engineers*, 1951, vol. 191, p. 540) it was observed that as the testing temperature was increased above the transition zone the energy for fracture of temper-embrittled steel approached a level different from that characteristic of unembrittled steel. Subsequent investigations, now reported, were concerned with the effects of hardness and temper-brittleness on the level of the energy *vs.* testing temperature curve above the transition zone. The steel studied was of the same composition as that used for the investigations reported in the previous abstract. A T.T.T. diagram developed by earlier work was available to indicate the extent of temper-embrittlement to be expected after a given austenitizing and tempering treatment: this diagram was used to select tempering treatments which would give variations in hardness, and a minimum degree of embrittlement.

The results reported lead to the conclusion that for steels of a given hardness the impact-energy level is higher for the unembrittled than for the temper-embrittled material. A linear relationship between impact-energy level and hardness exists in the hardness range Rockwell C-27 to at least C-38.

Nickel-Chromium-Molybdenum Steel Mining Drill Rods: Fatigue Properties

T. W. WLODEK: 'Effect of Water Corrosion and Shot Peening on Fatigue Strength of Mining Drill Rods.' *Canad. Mining and Metallurgical Bull.*, 1952, vol. 45, pp. 470-7; disc., pp. 477-8; *Trans. Canad. Inst. Mining and Metallurgy*, 1952, vol. 55, pp. 292-300.

The information contained in the paper is limited to the drill rod, as such, giving details of performance of the length of rod which lies between the bit attachment and the shank end, and is not usually exposed to the effect of forging and heat-treating operations carried out on the bit or the shank. The tests recorded were made by means of a simulated-service procedure: reversed bending was applied, under dry and under water-corrosion conditions.

Continuous investigation is in progress, in the mining and metallurgical industries, on field performance of different types of steels for drills, and the tests reported in this paper were designed to obtain additional basic information on the endurance strength and fatigue limit of two of the most commonly used steels, plain carbon type to A.I.S.I. 1080, and alloy steel containing carbon 0·47, nickel 2·93, chromium 0·37, molybdenum 0·21, per cent. Effect of water corrosion, on the fatigue strength of drill rods of the two steels in the as-rolled condition, was determined, and the effect of shot-peening was studied. Eight S-N curves relating to behaviour of the rods under dry and corrosive conditions are shown, indicating clearly the beneficial influence of such treatment. The highest endurance strength, under both conditions, was obtained in the alloy steel after peening.

Metallographic examination was made of the specimens after test, and the mechanism of failure in fatigue is discussed on the basis of the structures found and other observations made.

Influence of Boron on Case-Hardenability of Nickel-Alloy Steels

C. F. JATCZAK and E. S. ROWLAND: 'The Influence of Boron on Case Hardenability in Alloy Carburizing Steels.' *Amer. Soc. Metals, Preprint* 14, Oct., 1952; 13 pp.

The carburized end-quench test was used by the authors to evaluate the effect of boron on the case-hardening properties of low-alloy nickel-chromium and nickel-chromium-molybdenum steels (80B20, 94BV20, 94B20 and 47B20), over the carbon range 0·60-1·0 per cent., under several conditions of heat-treatment. Boron-free steels of substantially identical basis composition were similarly processed for comparison. Double- and single-quench treatments were simulated, by methods which are described.

The results showed that, for all the heat-treatments used, the effect of boron on case-hardenability of all the four steels decreased with rise in carbon content. In double-quench practice the effect of boron is low to negligible at high carbon levels when a normal hardening temperature (1475°-1525°F.: 800°-830°C.) is used. In general, at a given carbon level, the boron effect increased with rise in quenching temperature.

Both the single-quench treatments employed yielded much higher boron effects at the higher carbon levels than did the double-quench treatments at normal hardening temperatures. Both gave results about equal to those obtained on re-heating and quenching from 1700°F. (925°C.). No evidence was found of serious fading of the boron effect as a result of direct quenching, in steels of the carbon range investigated, and pot cooling to 1525°F. (830°C.) before quenching did not increase the boron effect. Particular attention is directed to effects observed in steels treated with Grainal.

HEAT- AND CORROSION-RESISTING ALLOYS

High-Temperature Materials for Gas Turbines: Symposium

IRON AND STEEL INST.: 'High-Temperature Steels and Alloys for Gas Turbines.'
Special Report 43, 1952; 395 pp.
Published by the Institute, July, 1952. Price £3 3s. 0d.

This volume contains the papers presented at the Symposium organized by the Iron and Steel Institute, in collaboration with other interested societies, in February, 1951. Discussions on the papers are presented (pp. 305-78) in groups corresponding to those in which the papers were given at the Symposium, and in this section is included the full form of a paper by W. SIEGFRIED, on 'Notch Sensitivity of Heat-Resisting Austenitic Steels', a shortened version of which was offered in discussion at the meeting.

For abstracts of the 36 papers presented at the Symposium see *Nickel Bulletin*, 1951, vol. 24, No. 6, pp. 116-28.

The Special Report also contains a reprint of the Fifth Hatfield Memorial Lecture, 'Some Problems in the Development of the Whittle Engine', delivered by AIR COMMODORE SIR FRANK WHITTLE, in association with the Symposium.

High-Nickel Alloys used in Aircraft

See abstract on p. 262.

Creep-Rupture and Recrystallization Studies on Monel

N. J. GRANT and A. G. BUCKLIN: 'Creep-Rupture and Recrystallization of Monel from 700° to 1700°F.'
Amer. Soc. Metals, Preprint 5, Oct., 1952; 20 pp.

The research reported in this paper is part of an extensive investigation in progress, at Massachusetts Institute of Technology, on the behaviour of metals and alloys under conditions of strain at elevated temperatures. The specific aims of this section of the work were:—

(1) To obtain creep-rupture data for one of the Monel alloys.
(2) To study the rôle of cold work on the creep-rupture behaviour of Monel.
(3) To relate, if possible, the recrystallization data for Monel with the creep-rupture behaviour.
(4) To confirm or adjust the suggested graphical method of extrapolating short-time creep-rupture data suggested by GRANT and BUCKLIN (*Trans. Amer. Soc. Metals*, 1950, vol. 42, pp. 720-61).

The material chosen was an alloy containing nickel 67, copper 30, iron 1·5, manganese 1, carbon 0·15, per cent., selected as being a single-phase non-age-hardenable alloy in which the known instabilities (under varying conditions of temperature, time, and strain) would be (*a*) transition from low-temperature type to high-temperature type behaviour; (*b*) recrystallization; and (*c*) oxidation. Other complicating structural or chemical instabilities were thus avoided, permitting easier realization of the objects listed in 1-4 (col. 1).

For determination of recrystallization behaviour, cold-worked bar stock was annealed for 30 minutes at 1500°F. (815°C.) and was then cold-worked, from 4 to 75 per cent. reduction of area, by cold-swaging. Time-temperature recrystallization data were then determined, using hardness, metallographic, resistivity and X-ray measurements: test times up to 500 hours were employed, to obtain extensive data. For the creep-rupture tests annealed material and two grades of cold-worked alloy were used (30 and 75 per cent.). The annealed specimens were also heat-treated for ½ hour at 1500°F. (815°C.) prior to testing, a treatment which, while not resulting in a dead-soft condition, kept the grain size within the desired limits. The creep-rupture tests were run for various periods from 0·001 to 2700 hours, at 700°-1700°F. (370°-925°C.).

The results, reported in extenso, confirm that the instabilities which adversely affect the long-time creep and rupture properties of Monel above certain temperatures are the incidence of intercrystalline fracture, oxidation and recrystallization.

Time-temperature relationships for recrystallization, as measured by hardness changes, do not give accurate indication of the onset of intercrystalline cracking in creep-rupture testing of Monel, and it is also found that in creep-rupture tests evidence of recrystallization is present at much lower temperatures and at shorter times than could be predicted from static recrystallization studies. Recrystallization occurs during creep-rupture testing of Monel, lowering creep-resistance and shortening rupture life, due to formation of very fine grain size along the grain boundaries.

It was found that 30 per cent. cold work effectively improves creep-rupture strength of the alloy up to 900°F. (480°C.) for a period of about 2000 hours. At temperatures above 1100°F. (595°C.) no benefit of the cold work was evident.

The data obtained confirm the validity of the method proposed by Grant and Bucklin for graphical extrapolation of short-time creep data.

Experimentally determined values of equicohesive temperature showed 700°F. (370°C) for a strain rate of 0·10 per cent. per hour for 30 per cent. cold-worked Monel of the grade studied.

Influence of Grain Size on High-Temperature Properties of Monel

P. SHAHINIAN and J. R. LANE: 'Influence of Grain Size on High-Temperature Properties of Monel.'
Amer. Soc. Metals, Preprint 6, Oct., 1952; 22 pp.

Influence of grain size of Monel was studied as likely to provide data generally valid with regard to the effect of this variable on strength and resistance to creep at high temperatures. The report of the experimental work is prefaced by a condensed but comprehensive review of the literature on the relation between grain size and creep behaviour, as found in various metals and alloys.

The wide range of grain sizes essential for the investigation (0·024-0·78 mm. average diameter) was produced by annealing as-received bars at various temperatures, or by cold working followed by annealing. In order to avoid interference by oxidation, treatments at temperatures above 1600°F. (870°C.) were carried out in vacuo. Full details of the annealing schedules are given, and the finest and coarsest structures are illustrated. Grain-size determinations were made on both the bar stock after final heat-treatment and on fractured specimens after creep test.

Creep tests were made at 700°, 900°, 1100° and 1300°F. (370°, 480°, 595° and 705°C.) and at stresses ranging from 7,000 to 65,000 p.s.i. (3 to 29 tons per sq. in.).

From the very extensive tests, results of which are reported individually in tabular form and by graphs, the following conclusions are drawn:—

'At low temperatures grain size has a pronounced influence on time-to-rupture and creep rate of Monel: increase in grain size produces shorter rupture times and higher rates of creep.

'At high temperatures there is an optimum grain size for maximum rupture time.

'There may or may not be an optimum grain size for the lowest minimum creep rate, depending on the temperature and stress. At 900° and 1100°F. (480° and 595°C.) there is an optimum grain size, but at 1300°F. (705°C.) the minimum creep rate decreases with increase in grain size.

'Influence of grain size on total elongation is small below the equicohesive temperature, but above that temperature total elongation generally increases with decrease in grain size. Above the equicohesive temperature the elongation of fine-grained Monel increases with rise in temperature.

'Reduction-in-area values vary with grain size in the same manner as total elongation. Generally, at low temperatures the per cent. reduction in area is higher than the per cent. total elongation, whereas at higher temperatures the per cent. total elongation is the higher.

'The influence of different heats, on rupture time and creep rate, is significant, particularly at the lower temperatures.'

Induction-Furnace Melting of Nickel and Nickel Alloys

See abstract on p. 260.

High-Temperature Properties of Wrought Chromium-Nickel-Cobalt-Iron-base Alloys

E. E. REYNOLDS, J. W. FREEMAN and A. E. WHITE: 'Influence of Chemical Composition on the Rupture Properties at 1200°F. of Wrought Cr-Ni-Co-Fe-Mo-W-Cb Alloys.' *Jnl. of Metals*, 1952, vol. 4, *Trans. Amer. Inst. Mining and Metallurgical Engineers*, pp. 946-58; T.P. 3344 E.

The paper records main features of the work covered by National Advisory Committee for Aeronautics Technical Note 2449, 1951; see abstract in *Nickel Bulletin*, 1952, vol. 25, No. 3, p. 73. The basis alloy used was a low-carbon type of N-155 Multimet.

Influence of Composition on Rupture-Strength of Chromium-Cobalt-Nickel-Iron Alloys

J. W. FREEMAN, J. F. EWING and A. E. WHITE: 'Influence of Chemical Composition on Rupture Test Properties at 1500°F. of Forged Chromium-Cobalt-Nickel-Iron-base Alloys.' *Nat. Advisory Committee for Aeronautics*, Tech. Note 2745, July, 1952: 69 pp.

The paper is one of a series reporting work conducted by the Engineering Research Institute of the University of Michigan, under the sponsorship of the National Advisory Committee for Aeronautics, as part of an investigation of the fundamental metallurgy of heat-resisting alloys used in propulsion systems for aircraft. The information is complementary to that given in an earlier report which covered the influence of composition on stress-rupture behaviour of the same types of alloy at 1200°F. (648°C.): N.A.C.A. Report 1058, 1951, which superseded N.A.C.A. Tech. Note 2449; abstract in *Nickel Bulletin*, 1952, vol. 25, No. 3, p. 73. See also preceding abstract.

The composition of the basic alloy and the ranges of alloy modification studied are shown in the table below:—

Basic Alloy	%	Ranges of Content Studied %
Carbon ..	0·15	0·08, 0·40, 0·60
Nitrogen ..	0·12	0·004, 0·08, 0·18
Manganese ..	1·7	0, 0·30, 0·50, 1·0, 2·5
Silicon ..	0·5	1·2, 1·6
Chromium ..	20	10, 30
Nickel ..	20	0, 10, 30
Cobalt ..	20	0, 10, 32
Molybdenum ..	3	0, 1, 2, 5, 7
Tungsten ..	2	0, 1, 5, 7
Niobium ..	1	0, 2, 4, 6
Iron	Balance	

In addition, molybdenum, tungsten and niobium were varied simultaneously in steps of 2 per cent., from 0 to 4 per cent. The amount of iron compensated for variation in total alloy content in all of the alloy modifications. In all, 62 modifications of the basis alloy were tested.

All the specimens were forged to bar stock, solution-treated at 2200°F. (1205°C.) for one hour, and aged at 1400°F. (760°C.). for 24 hours, using procedures designed to minimize effects of prior history. Rupture strengths for 100 hours and extrapolated values to 1000 hours were obtained. The conclusions drawn from the results, reported in extenso in the Note, are as follows:—

'1. The elements in the basic alloy can be varied individually over quite wide ranges without significantly affecting the rupture properties at 1500°F. (815°C.). Considerable reduction of nickel or cobalt could be tolerated without sacrifice in properties. Substantial

reductions of chromium, molybdenum, or tungsten would cause some reduction in strength. Increased carbon up to 0·40 per cent. would not change rupture strength but would reduce elongation in the rupture test. Increased silicon reduces strength. Increased niobium would be somewhat detrimental to strength. Omission of niobium reduces strength, except in the presence of increased amounts of molybdenum and tungsten. All niobium-free alloys have low elongation and large grain size.

'2. The 3:2:1 combination of molybdenum, tungsten, and niobium in the basic alloy is the optimum combination of these elements. Lower strengths result from lesser amounts and no improvement from increased amounts. Rupture strength equal to that of the basic alloy can be obtained in niobium-free alloys by increasing the molybdenum-plus-tungsten to about 8 per cent., although elongation will be much lower.

'3. There is little evidence to indicate that composition differences within the usual commercial limits contribute materially to scatter bands of properties observed in commercial production.

'4. Very little can be deduced from the data regarding the fundamental mechanisms by which composition controls properties. Some relationship between carbon, molybdenum, tungsten, and niobium seems to be a controlling factor in strength. A saturation effect reduces improvement from molybdenum and tungsten in the presence of niobium, but not in its absence. The increased elongation in the rupture test resulting from niobium additions appears to be due to grain refinement, reduction of intergranular precipitation, and influence on general precipitation characteristics.

'5. The results are similar to those at 1200°F. (650°C.) presented in N.A.C.A. Report 1058, except that increased chromium was not beneficial at 1500°F. (815°C.) and the saturation effect for molybdenum and tungsten did not occur at 1200°F. (650°C.).'

Properties of Austenite and Sigma

T. P. HOAR and K. W. J. BOWEN: 'The Electrolytic Separation and Some Properties of Austenite and Sigma in 18-8-3-1 Chromium-Nickel-Molybdenum-Titanium Steel.' *Amer. Soc. Metals, Preprint* 4, Oct., 1952; 19 pp.

Previous investigations of this type of steel have been concerned with the phases austenite, ferrite, and sigma as co-existing. The present paper describes the electrolytic separation of austenite and sigma, and a study of the chemical and physical properties of the individual phases. The steel used was of the following composition: carbon 0·04, manganese 0·43, silicon 0·56, chromium 18·4, nickel 8·45, molybdenum 3·40, titanium 0·47, per cent. Discs and rods of this steel, prepared from ½ to 1-in. diameter bar, were given a standard treatment at 1150°C., followed by water quenching and were then heat-treated for various times at 850°C. Austenite and sigma so formed were separated by selective anodic dissolution in 25 per cent. w./w. sulphuric acid at 0·4 amp./sq. in., and in 50 per cent. v./v. hydrochloric acid at 30 amp./sq. in., respectively. Experimental methods and equipment are fully described, and the structure of austenite and sigma produced under various conditions are illustrated.

Magnetic analysis of the extracts gave further evidence of the tendency of austenite to transform to α_2, and confirmed the observation previously made that sigma of certain compositions possesses a Curie point at low temperatures. Chemical analysis of the extracted phases showed that ferrite and (especially) sigma are richer in chromium and molybdenum, and poorer in nickel and iron, than the austenite with which they are associated. Sigma is shown to have a very low manganese content.

Electrode-potential measurements in sodium chloride and hydrochloric acid have demonstrated that sigma is usually more noble than the corresponding austenite in these electrolytes, and shows a greater tendency to remain, or to become, passive.

Formation and Effect of Sigma Phase in Commercial Iron-Nickel-Chromium Alloys

A. M. TALBOT and D. E. FURMAN: 'Sigma Formation and Its Effect on the Impact Properties of Iron-Nickel-Chromium Alloys.' *Amer. Soc. Metals, Preprint* 2, Oct., 1952; 12 pp.

The authors direct attention to the fact that most of the investigations of sigma formation in this group of alloys have been made on high-purity alloys or on alloys of compositions different from those which would normally be encountered in commercial materials today. The programme of the study reported in this paper was planned to permit close location of the sigma-phase area in the iron-nickel-chromium system, and to determine more exactly the maximum amount of sigma which may be present in an alloy of given composition without causing a serious degree of embrittlement. The compositions considered to be of main interest were 20/25, 35/15 and 35/20 nickel-chromium: the alloys used covered the range nickel 20-35, chromium 15-30, per cent. Incidental alloying elements were controlled within the following approximate limits: carbon less than 0·1, manganese 1·8, silicon 0·7 per cent.; sulphur and phosphorus were within limits normally present. High- and low-silicon variants of the 20/25 and 35/20 alloys were compared.

The material for examination was prepared in the following way: 10-lb. ingots were cast in 2 x 2-in. chill moulds, with core-sand hot tops which held about 25 per cent. of the metal. The head of the ingot was removed before forging and hot rolling to ¾-in. rounds. The ¾-in. bars were cold-rolled to ½-in. square bars, with an intermediate anneal, in order to obtain a final reduction of 23 per cent. Half of each heat was kept in the cold-rolled condition, and the rest was annealed for one hour (at temperatures chosen to give the same grain size, A.S.T.M. 4, in all heats), and water-quenched. The material from each alloy was thus available in three conditions: the cast structure was represented by the ingot head, and the wrought material was in the annealed and in the 23 per cent. cold-worked conditions.

Material in each of these conditions was exposed (for 100, 500, 1000 and 3000 hours) at 1200°, 1475° and 1650°F. (650°, 800° and 900°C.). Charpy keyhole-

notch impact values were obtained on the annealed and the cold-worked materials after the same periods of exposure, and hardness determinations were made on the broken halves of the Charpy specimens.

Identification of sigma was based chiefly on comparative metallographic examination: the most satisfactory etchant was found to be glycerin 3 parts, concentrated hydrochloric acid 2 parts, concentrated nitric acid 1 part. Details of etching procedure are discussed.

The results show that for conditions approaching equilibrium, i.e., exposure of cold-worked material to sigma-forming temperatures for 3000 hours, the limiting sigma boundary extends from 21 per cent. chromium at 20 per cent. nickel, to 24 per cent. chromium at 35 per cent. nickel. In the more stable and sluggish structures found in annealed or as-cast materials, the boundary was moved to somewhat higher chromium contents. Increase of silicon to 3 per cent. moved the sigma boundary to about 19 per cent. chromium with 25 or 35 per cent. nickel. It is noted that the sigma boundary as determined in this investigation is a reasonable extension of that found by NICHOLSON, SAMANS and SHORTSLEEVE for similar materials of lower alloy content (*Trans. Amer. Soc. Metals*, 1952, vol. 44, pp. 601-32).

Embrittlement, as measured by room-temperature impact properties after long exposure at sigma-forming temperatures, increased rapidly with the first few per cent. of sigma formed, regardless of the base composition of the alloy. Serious embrittlement may be produced by as low a sigma content as 5 per cent.

Carburization of 18-8 Steels

J. B. GIACOBBE: 'Mechanism of the Carburization of Some Stainless Steels.' *Amer. Soc. Metals, Preprint* 3, Oct., 1952; 15 pp.

In pack or solid carburizing it is generally believed that a gaseous phase, such as carbon dioxide or water vapour, which creates an 'oxidizing potential,' must be present in the solid, carbonaceous medium, in order that carbon transfer to the metal surface may occur. Under high vacuum, where the concentration of this gaseous phase would be extremely low, it has been reported that carburization of steels proceeds very slowly, if at all. In order to check the validity of the 'oxidizing potential' theory, the present authors undertook a series of experiments in which the 'carburizing' reaction was carried out under high vacuum, using high-purity graphite as the solid carburizer in contact with the metal surface. Because of their great affinity for carbon at temperatures above 1400°F. (760°C.), austenitic stainless steels of the 18-8 type were used, to permit detection of the slightest trace of carbon absorption: the tubing treated was of three types: A.I.S.I. 316, 347 and 304. A temperature of 1900°F. (1040°C.) was used for all the tests, as being the lowest generally employed in commercial annealing of these steels.

The observations made led to the conclusion that:—

'1. Austenitic chromium-nickel stainless steels of the 18-8 variety can absorb carbon from 'solid' carburizers, even though the partial pressure of carbon dioxide and water vapour is held to an extremely low value. The observed carbon absorption apparently cannot be explained on the basis of the 'oxidizing potential' theory.

'2. The rate of carbon absorption of this class of steels under a high vacuum is drastically reduced by the presence of an oxide film on the metal surface.

'3. Provided the surface oxide film of the stainless steel is decomposed or broken down under vacuum, carbon absorption, in the reduced gas phase, increases with an increase in contact pressure between the solid carburizer and the metal surface. As a result of this pressure, adsorbed carbon at the surface may react directly with chromium and molybdenum to form carbides which, under equilibrium conditions, provide the necessary carbon for entry into the austenite. The direct formation of carbides is attributed to the lowered energy of activation (as a result of surface or contact catalysis) for the formation of new compounds at the surface of the metal.'

Microconstituents in High-Temperature Alloys

H. J. BEATTIE and F. L. VERSNYDER: 'Microconstituents in High-Temperature Alloys.' *Amer. Soc. Metals, Preprint* 1, Oct., 1952; 27 pp.

By means of the complementary use of microstructural study and X-ray diffraction analysis of electrolytically separated residues, the authors investigated the microconstituents of typical complex iron-, cobalt-, and nickel-base alloys representative of materials used in high-temperature engineering. Full details of composition are given of the 18 materials used, and the techniques of examination are fully described.

The results show, in particular, some of the effects of titanium and molybdenum as alloying additions: the occurrence and suggested mode of formation of titanium carbonitride in a nickel-base age-hardenable alloy is dealt with at length. Conclusions applicable to most air-melted titanium-containing alloys are drawn.

The intermetallic compounds M_2Ti and Fe_2Al were identified and are discussed. A new phase, $CrMoN_x$ was observed and identified in a 16-25-6 chromium-nickel-molybdenum-iron alloy: its crystallography is described.

The second Brillouin zone is considered for the complex carbides M_6C and $M_{23}C$, with resulting determination of the ratio of electron to metal atom capacity of about 7:1 for these two carbides.

Nimonic Alloys in Power-Station Gas Turbine

'15 MW Gas Turbine Alternator Set at Trafford Power Station.' *Engineer*, 1952, vol. 194, Sept. 12, pp. 343-5.

The article describes, with many illustrations, the first gas-turbine alternator set to be commissioned by the British Electricity Authority, which went on load at Trafford Power Station, Manchester, on August 6th, 1952. Details of materials used in various parts of the plant include reference to rotor blading of Nimonic 80A and the use of Nimonic 75 and austenitic nickel-chromium steels for other components.

High-Temperature Properties of 18-8 Chromium-Nickel Steels

E. J. DULIS, G. V. SMITH and E. G. HOUSTON: 'Creep and Rupture of Chromium-Nickel Austenitic Stainless Steels.' *Amer. Soc. Metals, Preprint* 7, Oct., 1952; 35 pp.

The experiments were an extension of work carried out by the authors on Types 304, 321, 316 and 347 steels (*Trans. Amer. Soc. Metals*, 1950, vol. 42, pp. 935-80). Creep- and rupture-test data are presented for A.I.S.I. Types 304L, 304L (high-nitrogen), 316L, 316-Nb and 303. Additional data on 304, 321 and 347 are also included. Changes observed in microstructure after high-temperature test, and final mechanical properties at room temperature are recorded, together with data on magnetic permeability, at room temperature, before and after high-temperature exposure.

The principal findings are as follows:—

1. With regard to reproducibility of properties, excellent agreement was obtained with Type 304, slight scatter was observed with Type 347, and Type 321 showed relatively wide scatter in creep rate, but only slight scatter in creep-rupture strength. These conclusions are subject to confirmation after investigation of additional heats.

2. The previous studies had indicated but little difference between annealed and stabilized Type 321, or annealed and stabilized 347. In the later tests, however, Type 321 steel was slightly inferior in the stabilized condition. Stabilization heat-treatment of 316-Nb slightly lowered creep-rate strength at 1300° and 1500°F. (705° and 815°C.), and creep-rupture strength at 1300°F. (705°C.).

3. The E.L.C. grades of Types 304 and 316 proved significantly weaker in creep than the corresponding standard-carbon grades. Nitrogen additions to 304L raise the strength at 1100° and 1300°F. (595° and 705°C.) to a level above that of standard 304, but at 1500°F. (815°C.) nitrogen is ineffective.

4. No general statement can be made, on the basis of the results obtained in these tests, regarding the relative strengths of Types 321 and 347, other than that 347 appears to be superior in the intermediate temperature range, i.e. about 1100°F. (595°C.).

5. Free-machining 18-8 (Type 303) and Type 304 have substantially the same creep strengths at 1100°, 1300° and 1500°F. (595°, 705° and 815°C.).

6. Study of the relative effects of creep exposure on notch-impact strength, hardness, and tensile properties showed impact strength to be most severely affected.

7. Sigma formation and precipitation of carbide were the principal microstructural changes observed. Sigma formed most rapidly (even during stabilizing heat-treatment) and most plentifully (up to 15 per cent.) in Type 316-Nb. In contrast, one of the 321 steels showed no sigma after stabilization treatment, even though it contained δ ferrite and some δ ferrite had apparently persisted untransformed throughout the 3000-hour creep test. Sigma was observed in all the low-carbon grades, but it probably formed from the δ ferrite.

Resistance of Nickel-Chromium-Iron Alloy to Steam

P. OGDEN and R. ACORAH: 'The Effect of High-Temperature Steam on a Nickel-Chromium-Iron Alloy.' *Univ. Missouri, Engineering Experiment Station Bull. Ser.*, 1952, vol. 53, No. 38.

The report describes extensive experiments made to determine the effect of high-temperature steam attack (at 2200°F.: 1205°C.) on a nickel-chromium-iron wire (nickel 57 min., chromium 14-18, manganese 3 max., silicon 0·75-1·5, carbon 0·25 max., sulphur 0·03 max., per cent., iron remainder).

The results showed that at the surface high-temperature steam attack results in formation of a thin, hard film of chromium oxide which has properties specially useful in the field of steam generation. In the interior of the alloy high-temperature steam attack leads to chemical reactions with metal oxides, carbides, etc. These reactions may weaken and open up the grain boundaries, cause local melting and recrystallization, induce high thermal stress, generate gas at high pressure, and even extrude plastic material from the interior through the surface. These effects become increasingly critical as the operating temperature approaches the melting point of the alloy.

Electron-Diffraction Investigation of Austenitic Nickel-Chromium Steel

J. F. BROWN and D. CLARK: 'The Use of the Three-Stage Electron Microscope in Crystal-Structure Analysis.' *Acta Crystallographica*, 1952, vol. 5, Sept. 10, pp. 615-19.

The combination of electron diffraction and electron microscopy in a three-stage electron microscope enables electron-diffraction patterns to be obtained from extremely small single crystals, i.e., less than 1μ across and 1000 Å thick. Two examples are described, in which it is shown how these single-crystal diffraction patterns can be used to obtain valuable crystallographic information on unit-cell size and symmetry. Examples used relate to investigation of two austenitic steels, containing, respectively, chromium 18, nickel 8, molybdenum 2, titanium 1, per cent., and chromium 25, nickel 20, per cent. These steels were studied after heat-treatment in the range 650°-850°C., for periods up to 1000 hours, with particular reference to the growth of the sigma phase and precipitation of carbides. Eight illustrations demonstrate the results obtained on these steels by the technique described, and an X-ray powder pattern of sigma phase from the complex 18-8 steel is also shown.

Conservation of Nickel in Stainless Steels

'Substitution of Straight-Chromium Stainless Steels for Chromium-Nickel Types.' *Metal Progress*, 1952, vol. 62, Sept., pp. 97-114.

A symposium, consisting of short contributions from executives and research workers, critically evaluating the current situation, and the properties of the two types of steel as affecting possibility of interchange, and discussing some of the applications for which the straight-chromium steels can or cannot be used in place of the austenitic steels.

Austenite-Martensite Transformation in Stainless Steels

G. H. EICHELMAN and F. C. HULL: 'The Effect of Composition on the Temperature of Spontaneous Transformation of Austenite to Martensite in 18-8-Type Stainless Steel.' *Amer. Soc. Metals, Preprint* 9, Oct., 1952; 19 pp.

Although maintenance of an entirely austenitic structure is in many cases desirable in stainless steels, austenite instability may sometimes have beneficial effects, e.g., austenitic steels in which the austenite transforms to martensite during rolling or wire-drawing at sub-zero temperatures attain unusually high tensile strength, particularly if the material is aged at 750°F. (400°C.) after deformation.

Studies of austenite stability reported in the literature have been confined mainly to investigation of the effect of composition on formation of ferrite, corrosion-resistance, the phases present, and transformation induced by deformation. In spite of the commercial and theoretical significance of the M_s temperature of stainless steels, no data have been published on this aspect.

A practical objective of the research reported in this paper was to provide a method for calculating the M_s temperature of a stainless steel from its composition, thus facilitating selection of steels for use under specific conditions and for applications in which particular combinations of properties are required. The steels studied were of six different types:—

(1) 12% chromium with varying nickel;
(2) 14% chromium with varying nickel;
(3) 17% chromium with varying nickel;
(4) 12/10 and 17/7 chromium-nickel with varying carbon;
(5) 12/10 chromium-nickel with varying manganese;
(6) 10/12 chromium-nickel with varying silicon.

Effect of composition on temperature of the martensitic transformation during cooling was determined by dilatometric measurements. The results show that the relative effectiveness of the elements in lowering the M_s temperature increases in the following order: silicon, manganese, chromium, nickel, and carbon or nitrogen. The effect of the alloying elements, in weight per cent., on the M_s temperature (in °F.) can be calculated approximately by the equation:—

$$M_s = 75(14{\cdot}6 - Cr) + 110(8{\cdot}9 - Ni) + 60(1{\cdot}33 - Mn) + 50(0{\cdot}47 - Si) + 3000(0{\cdot}068 - [C - N])$$

The nickel equivalents of silicon, manganese, chromium, carbon and nitrogen are 0·45, 0·55, 0·68, 27 and 27 respectively.

Precipitation of carbides results in an increase in the M_s temperature, which varies with the amount of carbon and chromium removed from solid solution. M_s changes caused by carbide precipitation can be used as a sensitive means for determining the solubility of carbon in stainless steels.

Resistance of Metallic Materials to Attack by Hydrogen Peroxide

N. S. DAVIS and J. H. KEEFE: 'Equipment for Use with High-Strength Hydrogen Peroxide.' *Jnl. Amer. Rocket Soc.*, 1952, vol. 22, Mar.-Apr., pp. 63-9.

High-strength hydrogen peroxide is in wide use to-day as a monopropellant and as an oxidant in bi-propellant rocket systems. This paper reports some results of research on materials suitable for handling concentrated hydrogen peroxide in storage and propellant equipment.

A brief review is made of factors governing selection: (1) effect of the material on rate of decomposition of the H_2O_2; (2) effect of the H_2O_2 on the materials; and (3) the possibility of formation of detonatable mixtures with H_2O_2. The authors then report results of laboratory tests made on metallic and non-metallic materials, classifying the data according to recommended use under long- or short-time contact, and listing those entirely unsuitable for use in any conditions. The nickel-chromium stainless steels, Durimet, and the Hastelloy alloys, are graded as suitable for short- but not long-time contact. Copper, lead, iron or carbon steel, Inconel, Monel and Stellite No. 6 are considered unsuitable for handling H_2O_2 under any conditions studied. Aluminium alloys are generally very useful.

A later section of the paper is concerned with design of equipment, emphasizing details by which life can be lengthened and efficiency increased. Finally, typical items of equipment are described and illustrated. Those mentioned include several in which stainless-steel components are incorporated.

Corrosion-Resisting Nickel-Steel Tubing in Oil-Condensate Wells

T. S. ZAJAC: 'Gas-Condensate Well Corrosion: What It Costs and How to Combat It.' *Oil and Gas Jnl.*, 1952, vol. 51, Aug. 11, pp. 108, 110, 112.

This paper, prepared by a member of the Shell Oil Company, New York, is described as 'a summary of what is being learned about this costly problem, what causes it, how to detect and evaluate it, and the techniques and measures for controlling it.' The report was presented at the 1952 meeting of the National Gasoline Association of America. The Corrosion Research Project Committee of the Association is making a compilation of all pertinent information on this subject, which is to be issued in handbook form, probably during 1953.

The present summary critically reviews information on the costs of uncontrolled corrosion, the costs of chemical treatments, and the use of corrosion-resistant materials (such as 5 and 9 per cent. nickel steel or nickel-plated steel) in tubing, with emphasis on the economic aspect of the use of special materials. An estimate is made of savings which might be effected by introduction of adequate corrosion-control measures.

Corrosion-Resisting Materials in Marine Engineering

L. W. JOHNSON and E. J. BRADBURY: 'Corrosion-Resistant Materials.'
Mond Nickel Co., Ltd., Publn. 588; 39 pp.*

The publication is based on the paper presented to the Institute of Marine Engineers, Feb., 1951; see abstract in *Nickel Bulletin*, 1951, vol. 24, No. 6, pp. 144-5.

* *We shall be pleased to supply a free copy of this publication.*

Hastelloy 'D': Reference Data

'Hastelloy Alloy D.' *Chemical Engineering*, 1952, vol. 58, Aug., pp. 258, 260, 262, 264, 266.

This is a further item in the series giving information on the behaviour typical of certain materials in contact with specific corrosives. For reference to data sheets on Hastelloy C and B see *Nickel Bulletin*, 1952, vol. 25, No. 10, p. 243.

The composition range of Hastelloy D is given as silicon 8·5-10·0, copper 3·85-4·25, manganese 0·80-1·25, carbon 0·12 max., chromium 1 max., iron 1 max., per cent., nickel balance. The most outstanding application of this alloy is for equipment used in concentrating sulphuric acid, and for handling sludges containing this acid, the corrosion-resistance of the alloy being due to formation of a protective sulphate film.

As in earlier summaries in this series, the information on the behaviour of Hastelloy D in contact with various corrosive substances is presented in the form of charts indicating degree of resistance in four qualitative classifications. Typical mechanical properties and hardness data are also given.

Etching of Stainless Steel

'Etching Processes.' *Pera Bull.*, 1952, vol. 5, Mar., pp. 95-7.

The Production Engineering Research Association announces its intention to summarize, from time to time, information which has been compiled in connexion with individual enquiries but which is of interest to a wider public. This summary on etching processes is an example. It contains practical notes on (1) etching chromium-plated steel and stainless steel, (2) black etch on steel, (3) photographic etching, and (4) an acid-etching selective method used in Germany for marking of tools. With regard to etching of stainless steel, the following solutions are mentioned:—

(1) Ferric chloride 20 g., hydrochloric acid 20 g., water 60 c.c.
(2) Saturated solution of ferric chloride in water: treatment for about 3 minutes at r.t.
(3) Ferric chloride 20 g., hydrochloric acid 25 c.c., nitric acid 5 c.c.,+ water (general production etching solution).
(4) Hydrochloric acid 4 parts, nitric acid 3 parts, water 4 parts (etches rapidly but reaction not easy to control).

It is noted that in all cases wetting agents are usually added. Solutions (1) and (2) are apt to cause localized pitting: (3) is recommended as most generally reliable.

A simple paraffin coating may be used to protect the surface of the steel from acid attack: this can subsequently be removed, and the metal passified by immersing the etched surface in warm 20 per cent. nitric acid for 30 minutes, after which it should be rinsed and subjected to a neutralizing treatment in an alkaline solution.

Stainless-Steel Parts made by Powder Metallurgy.

'Stainless Steel used to Advantage in Powdered Form.' *Nickel Topics*, 1952, vol. 5, No. 6, p. 6.

The availability of pre-alloyed stainless-steel powder is now making possible combination of the corrosion-resistance of stainless steels with the special properties and economic advantages of the powder-metallurgy process. Filters made from molybdenum-containing nickel-chromium steel powder have already solved some problems in the food, petroleum and chemical industries, and, for use in conditions of combined high-temperature and corrosion attack filter units are being made in special nickel- and cobalt-base alloys. Self-lubricating bushings are a further appliication of the pre-alloyed stainless powders, and reference is also made to their use in water-meter parts. Some typical items produced from pre-alloyed powders are illustrated.

Testing of Corrosion-Resisting Materials

E. G. HOLMBERG: 'Corrosion Testing. A Tool of Conservation.' *Chemical Engineering Progress*, 1952, vol. 48, Aug., pp. 377-80.

The author opens with a discussion of the relative reliability of some of the methods which may be used for assessing the suitability of metallic materials for use under specific corrosive conditions. He then describes methods which have been successfully used (by Alloy Steel Products Company, Linden, New Jersey) in determining which alloys are most suitable for use under typical conditions. One of the corrosion-test assemblies in current use is illustrated, with notes on methods adopted for prevention of galvanic and other effects which might vitiate the reliability of the data obtained. A test period of 60 days is usually employed.

Typical corrosion-test results are given relating to some representative alloys used in chemical-engineering plant, e.g., stainless steels of the nickel-chromium and nickel-chromium-molybdenum types, copper-base alloys Monel, nickel-molybdenum-base alloys, and nickel-silicon-copper alloys. The influence of addition elements, on some of these basic compositions, is discussed, and results of corrosion tests in various solutions are tabulated. The information given includes data on behaviour in the following corrosives: (1) solution containing acetic acid, methyl formate, acetaldehyde, methyl acetate, ethyl acetate, acetone, methyl alcohol, and formic acid; (2) 0·5-1% sulphuric acid + small amounts of ammonium sulphate; (3) saturated ammonium sulphate + 2% sulphuric acid; (4) 10% sulphuric acid without impurities; (5) solution containing nicotine sulphate, nicotine and sulphuric acid; and (6) solutions containing small amounts of sulphuric acid and ammonium sulphate+an unknown contaminant. Detailed comments are made on the resistance of the various alloys tested, as a function of basis composition and of modification by small amounts of other elements.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

PRINTED IN ENGLAND *by* WIGHTMAN MOUNTAIN LTD.
WESTMINSTER, LONDON, S.W.1